Exploring the Particle Reconfiguration in the Metallic Materials under the Pulsed Electric Current

Journal Item

Published at ***Steel Research International***, DOI: 10.1002/srin.201800062. (2018)

Exploring the particle reconfiguration in the metallic materials under the pulsed electric current

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Abstract

The dissolution, refinement and separation of the particles are one of the core metallurgical processes to control the quality of metallic materials. The dimensional changes and controlled motion of the particles have attracted great attention due to their important contribution to the corrosion resistance, service life and mechanical properties of metallic materials. In general, the dimensional change of the precipitates is controlled by heat treatment, while the separation or modification of the inclusions is completed by means of filtration, argon blowing and Ca addition. Sometimes, these methods are inefficient and can not meet the in-situ operation requirement in a particular working environment. This study aims to introduce a new idea of electropulsing metallurgy to achieve direct control for inclusions and precipitates, and this technique is efficient and green. The production of high-quality steels is achieved by the refinement and separation purification of the inclusions. The regeneration of the aged stainless steel used in nuclear power plant is obtained by the dissolution of precipitates. Therefore, electropulsing metallurgy provides a new technique to control the particle morphology and meets the needs for clean smelting purification and high-performance metallic materials fabrication. Scientific understanding of electropulsing processing, however, is limited. Some difficulties are highlighted here if the field is to make substantive progress.

Keywords: Electropulsing; refinement; segregation; separation; dissolution; particle

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1. Introduction

Particle reconfiguration has drawn great attention due to its tremendous use in metallurgy, materials and medicine.[1-3] In general, energy injection is a prerequisite for initiating particles movement or making the particles refinement. Applying an external field, which can be magnetic,[4] electric,[5] UV light,[6] thermal,[7] mechanical,[8] and microwave[9], is one way to manipulate particles. Of these, electric field is especially convenient for practical purposes since their magnitudes, phases, and frequencies can easily to be adjusted. Here, we consider a low-conductivity solid particle in a highly conductive matrix, of course, the matrix can be liquid or solid, if an electric current is applied to the system, how the morphology and location of particles will change? Based on thermodynamics, the system free energy in this situation consists of chemical free energy G_c, interfacial energy G_i, strain-stress energy G_s and electric current free energy G_e. Thus, the system free energy change (ΔG) in microstructural evolutions is represented as[10]

$$\Delta G = \Delta G_c + \Delta G_i + \Delta G_s + \Delta G_e \tag{1}$$

The microstructural evolutions are determined by the irreversible law of thermodynamics

$$\Delta G \le 0 \tag{2}$$

For the system without electric current, that is, $\Delta G_e = 0$, microstructural evolutions happen when $\Delta G_c + \Delta G_i + \Delta G_s \le 0$. For the system with electric current, the particle manipulation takes place when $\Delta G_e > 0$. The term G_e is obtainable from the expression of[10,11]

$$\Delta G_e = \frac{\mu}{8\pi} \iint \frac{\vec{j}_b(r)\vec{j}_b(r') - \vec{j}_a(r)\vec{j}_a(r')}{|r - r'|} dr dr' \tag{3}$$

where μ is the magnetic permeability. $\vec{j}_b(r)$ and $\vec{j}_a(r)$ are current density distributions before and after the applying electric current, respectively. r and r' are two different positions inside the system. The calculation of ΔG_e requires the distribution of current density. Normally, numerical calculation can be performed to give the quantitative value of ΔG_e, but the calculation is a complex process. In essence, ΔG_e is proportional to a geometric factor *k*, to the square of the current density j^2 and to $\frac{\sigma_1 - \sigma_2}{2\sigma_1 + \sigma_2}$. The general expression of ΔG_e can be expressed as follows[12,13]

$$\Delta G_e \propto \frac{\sigma_1 - \sigma_2}{2\sigma_1 + \sigma_2} k j^2 \tag{4}$$

where σ_1 and σ_2 are the electric conductivities of the matrix and particle, respectively. The geometric factor *k* is positive, and is associated with the dimensions of the matrix and particle. The experimental and calculated results show that the electric current promotes a structural evolution in the material towards the state with overall lower electrical resistance. The configuration (e.g. morphology, orientation and location) of the particles in the metal matrix will affect the geometric factor *k* and produce an arrangement towards a certain direction so that the electrical resistance along the electric current direction is at minimum. From this, it can be inferred that both the morphology and the location of the particles will evolve to meet the requirement of decreasing the free energy of the overall system. The first case can be seen from **Figure 1**, that is, the particle with high electrical resistivity is pushed from the position of Figure 1a to the position of Figure 1b. This situation can be used for the inclusions separation and liquid purification, as well as to study the segregation behavior of atoms. In another case from **Figure 2**, the particle in Figure 2a is refined into more small particles in Figure 2b to minimize the electrical resistance of the system. This can be used for dissolution of precipitates, refinement of inclusions and phases.

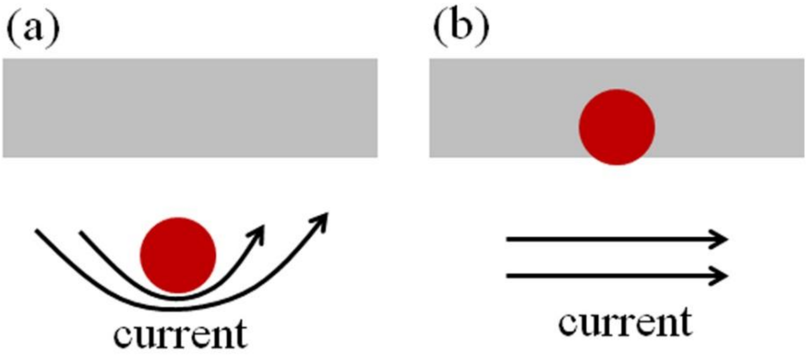

Figure 1. Particle motion from (a) to (b) under electric current

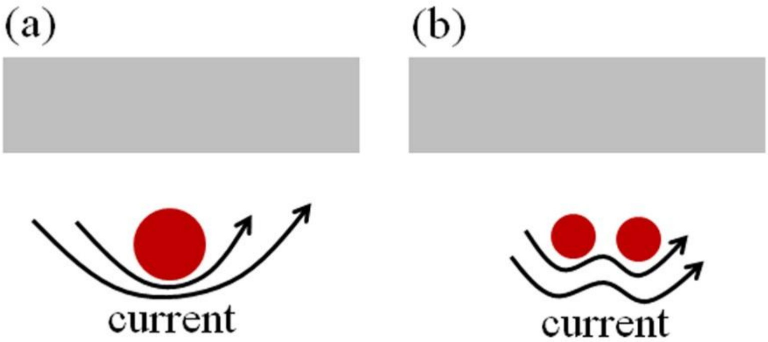

Figure 2. Particle refinement from (a) to (b) under electric current, (a) and (b) have the same volume.

In fact, a large power injection is also very energy-consuming. Pulsed electric current, as an instantaneous high energy input method with high efficiency and low energy consumption, is effective to solve this problem. It would be exciting to imagine that novel structures in the solid state can be induced in milliseconds, that materials exposed to service can be regenerated in similarly short times, and the molten metals can be purified by short duration treatments.[14,15]

Our aim was to introduce an extra free energy term into the system so that the particles configuration can be controlled. However, scientific understanding of electropulsing processing is limited. Some difficulties are also highlighted here if the field is to make substantive progress.

2. Parameters of pulsed electric current

Electropulsing refers to a pulsed electric current with current density >10^3A/cm^2. The peak current density, wave shape, pulse frequency, pulse duration, loading time and loading method will be designed elegantly to accomplish various tasks. In general, the effect of current density on the microstructure is the most significant, that is, the larger the current density, the easier the microstructure modification. However, the wave shape affects the average current density. At the same amplitude (e.g. the peak current density), the average current density of the square and sine waves varies greatly. The pulsed frequency (>200 Hz) contributes the most to Joule heating, while severe skin effects occur at high frequencies, which can be detrimental to the modification of the internal microstructure in the metals. The pulse width directly affects the current application time on the metals. The pulse with large duration results in high Joule heating. Loading time will be adjusted according to the characteristics of the investigated object, ranging from milliseconds to few hours. The loading method greatly affects the current distribution, thereby affecting the current distribution around the particles. Therefore, in order to obtain the best processing technology these parameters must be optimized. In addition, the effect being studied here is different to that from ohmic heat, electromagnetic levitation, pinch or skin effects. Schematic diagram of the pulsed electric current acting on the metals is shown in **Figure 3**.

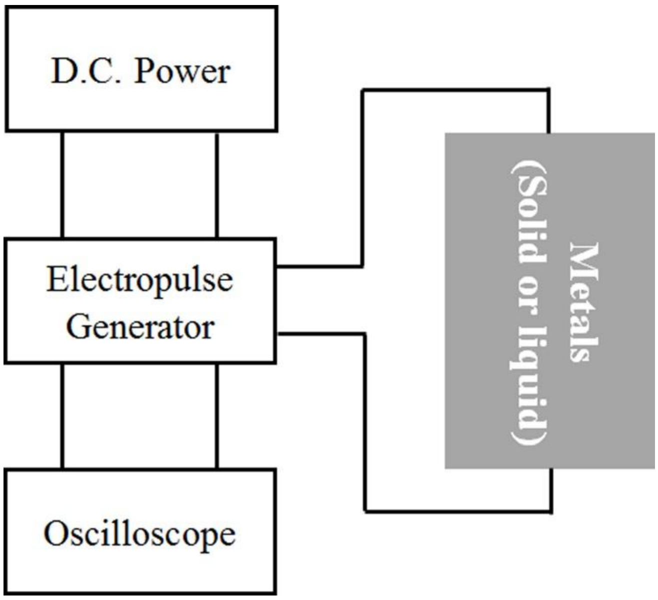

Figure 3. Schematic illustrations for electropulsing processing

3. Research progress in particle reconfiguration

3.1 Inclusions separation from molten steel under pulsed electric current

Non-metallic inclusions are detrimental to the mechanical properties and the casting reliability of clean steels.[16] Large inclusions (>20 μm) can be extracted from molten steels by electromagnetic stirring[17,18], bubble[19] and filtration[20], but the inclusions less than 20 μm are extremely challenging for separation because of the combination of Brownian motion and strong viscous force[21]. At present, a controlled motion of electrically neutral inclusions in a conductive molten steel at high temperatures has been realized under the pulsed electric current.[22-25] It can be seen that the Al_2O_3-MnS inclusions were distributed uniformly in the steel matrix in the as-solidified untreated steel (**Figure 4a**). The distribution of the Al_2O_3-MnS inclusions was completely different in the electropulsed steel (**Figure 4b**). It is obvious that the Al_2O_3-MnS inclusions disappeared from the inner part of the steel matrix, and instead were dispersed at the surface of the steel. It is suggested that electric current expels a high resistivity object (inclusion) from a low resistivity matrix (steel).

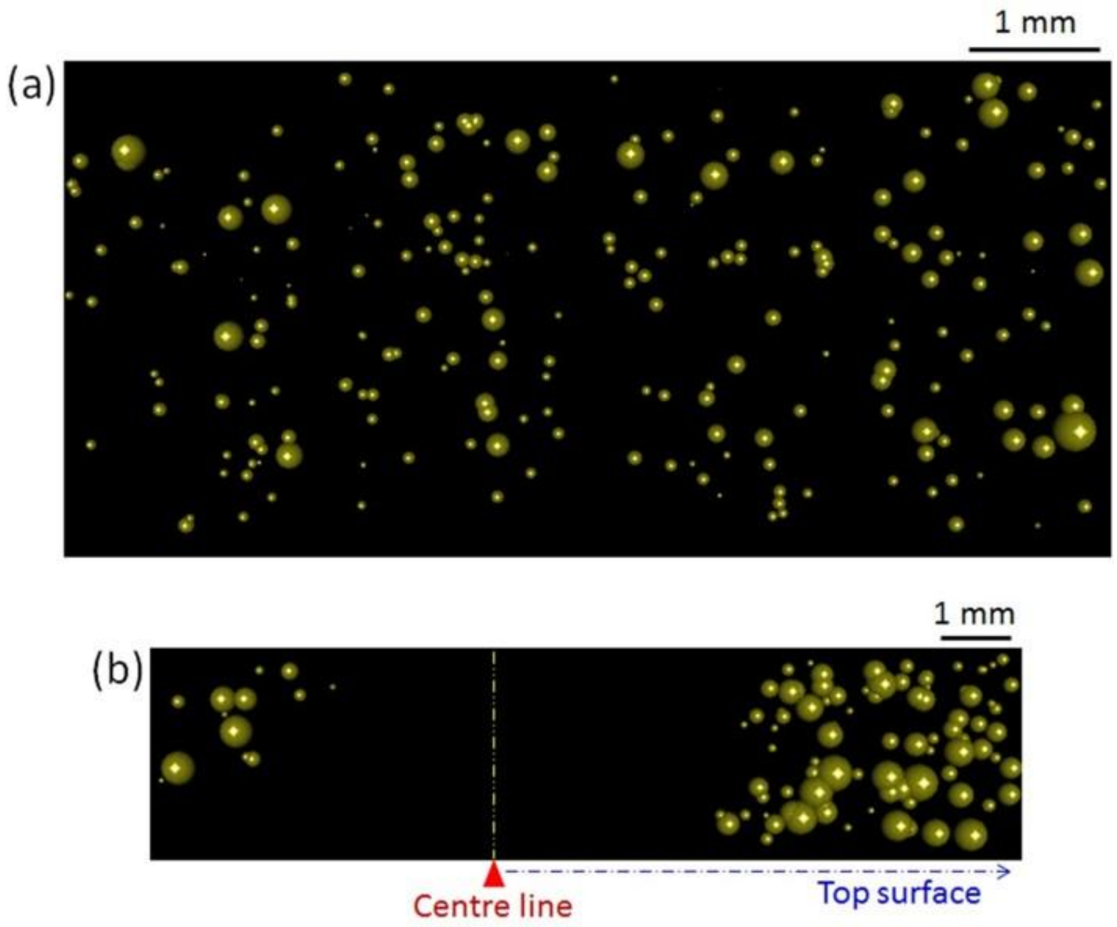

Figure 4. The number distribution of Al_2O_3-MnS inclusions in the untreated- and treated-steels. Automated particle analysis by FEG-SEM was applied to detect the distribution of inclusions in the molten steel. The pulsed electric current was applied in square-wave form. The frequency of the pulse is 1 Hz and the duration of each pulse is 60 μs. The density of the pulsed electric current was $1.6\times10^6 A/m^2$. The total treatment time is 8 minutes (including the solidification time). (a) Untreated steel, (b) Electric-current-treated steel.

In order to further clarify this separation behavior, the inclusions are implanted in a strong convection environment. We blow large argon bubbles (11mm) from the bottom to create this convection effect. The results indicate that strong convection has little effect on the re-distribution of inclusions under the pulsed electric current (**Figure 5**).

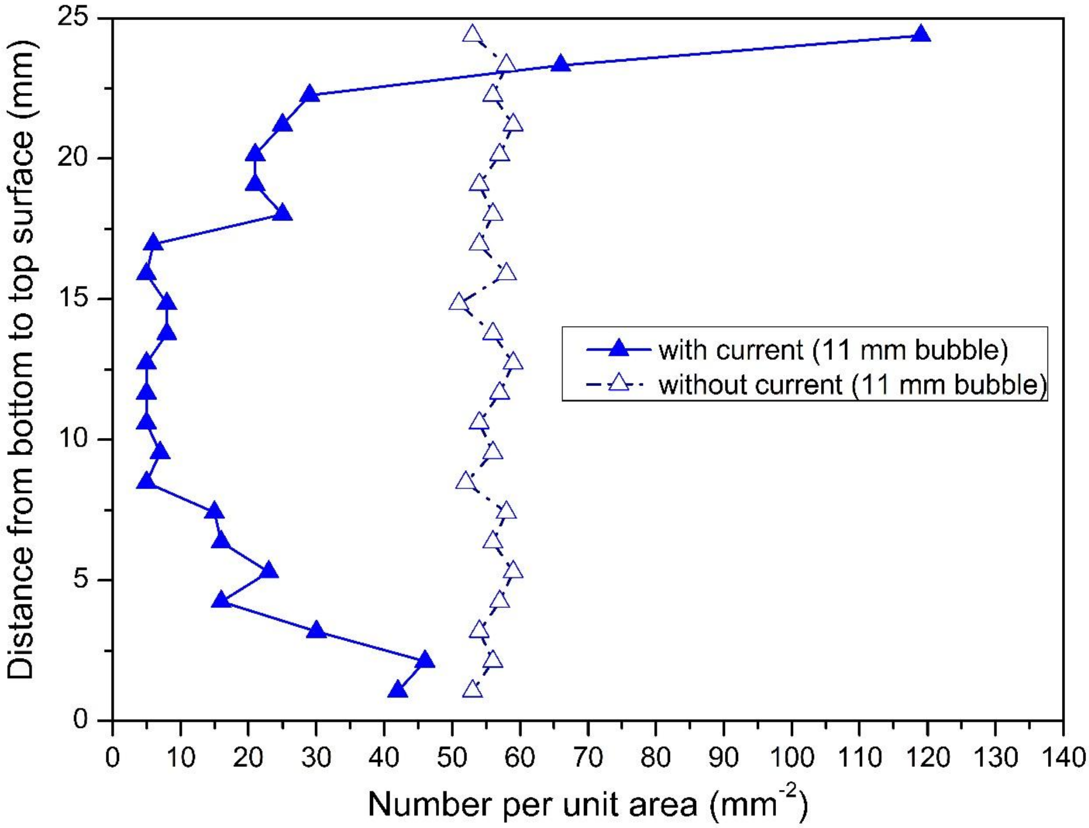

Figure 5. Number distribution of inclusions in liquid metal without and with electric current treatment. The inclusions disappear from the inner part of the steel matrix and instead are dispersed on the surface (e.g. double well shape).

Electrically neutral particles in liquids are separated according to the discrepancies of their electrically conductivities. In general, the conductivity of the liquid has a value of 10^5 $\Omega^{-1}m^{-1}$ and that of Al_2O_3 above 1673 K is approximately 10^{-2} $\Omega^{-1}m^{-1}$.[22,26] Thus the value for Al_2O_3 particles is 10^7 times smaller than that of the liquid. On the basis of Equation (4), it can conclude that the inclusions will be driven from liquid by the electric current in order to minimize the system energy. In addition, the velocity of the particles with electric current is proportional to particle volume, pulsed electric density, and the particle position, while that of particles without electric current is only related to their sizes.[22]

3.2 Precipitates dissolution under pulsed electric current

Coarsening of precipitates can give rise to the loss of strength, localization of stress, initiation of crack, creeping and many other detrimental effects. This takes place frequently in engineering alloys implemented at an elevated temperature especially after long durations.[27] Normally, the precipitates have different electrical conductivities from the steel matrix. The different configurations of the precipitates in steel affect the electrical current distribution in the whole system (**Figure 6**). It can be seen from Figure 6a, the current distribution will be uniform in the case of a particle with conductivity equal to that of the surroundings. In contrast, the distribution is markedly different when the conductivity of a particle is lower than the environment (Figure 6b). Electrical processes on the surface of the particle tend to change the current distribution from the one pictured in Figure 6b to that of Figure 6a.[22] This is driven by the tendency to reduce the system free energy. It is suggested that electric current may affect the formation of object with different electrical conductivities from that of matrix. This may provide a possibility to use electric current to affect the formation of precipitates.

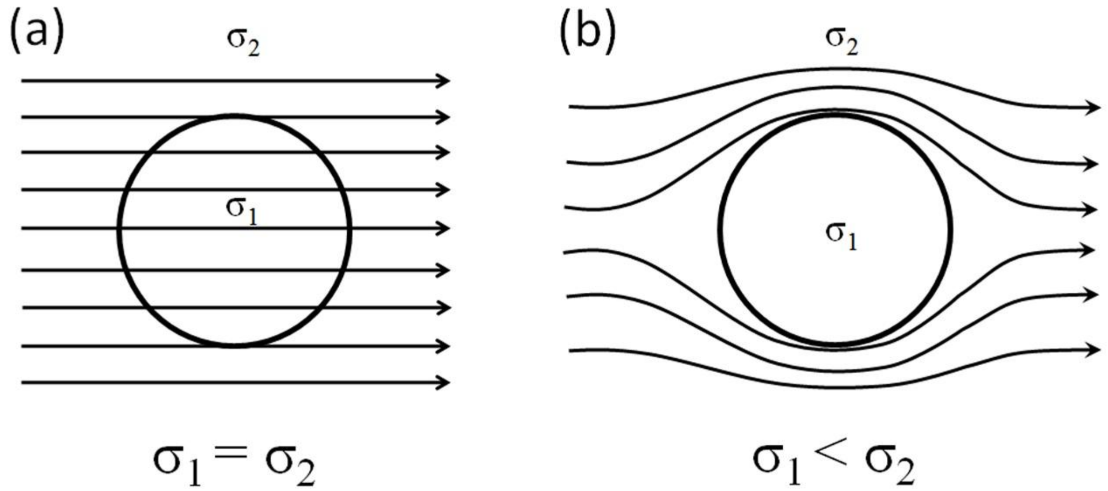

Figure 6. Refraction of electric current lines. Electric current distribution for a particle with conductivity equal to that of its environment in Figure 6a, and for a particle of lower conductivity in Figure 6b. σ_1 is the conductivity of particle, and σ_2 is the conductivity of the matrix. Reproduced with permission.[22] Copyright 2015, Springer Nature, Macmillan Publishers Limited.

χ-phase ($Fe_{36}Cr_{12}Mo_{10}$) is an intermetallic compound that is frequently found in austenitic stainless steels underwent a long-time treatment at the moderate temperature. **Figure 7**a illustrates the distribution of precipitates in the sample without electropulsing treatment. The particles are distributed randomly in the matrix, that is, a high frequency of precipitates was observed both along the grain boundaries and within the grains. However, the average size of the precipitates in the electropulsed steels is much smaller than that of without electropulsing treatment (Figure 7b). It indicates that the precipitates are dissolved by electropulsing significantly.[28] Similarly, experimental results also show that the dissolution rate of β-$Mg_{17}Al_{12}$ phase in aged Mg-9Al-1Zn alloy can be enhanced by pulsed electric current.[29,30]

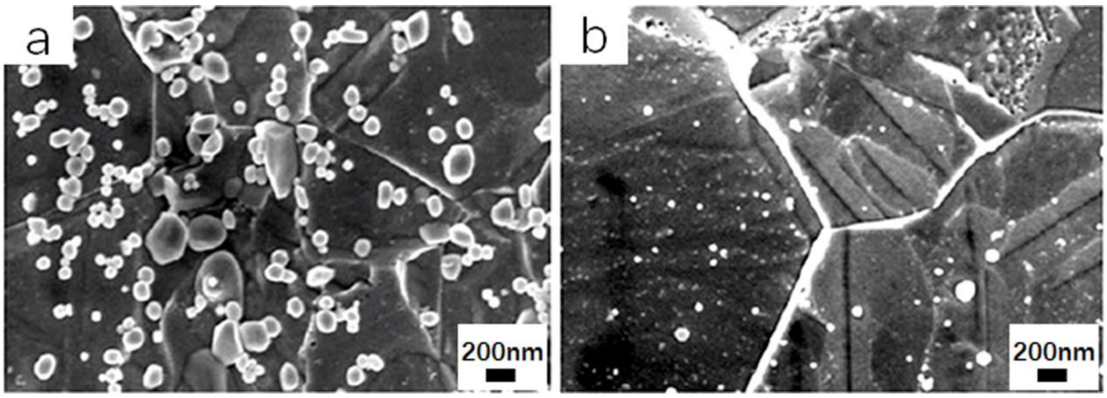

Figure 7. Distribution of precipitates in 316L stainless steels in the untreated- and treated-samples. (a) for the untreated sample, and (b) for the electropulsed sample. Reproduced with permission.[28] Copyright 2015, the Institute of Materials, Minerals and Mining (IOM^3), Taylor & Francis Group.

3.3 Electropulsing-induced elemental segregation

Segregation of a solute to surface or an internal interface of a solid produces a material with a discrete composition and its own set of properties that can have important (and often deleterious) effects on the overall properties of the materials. These zones with an increased concentration of solute can lead to interface fracture as a result of creep embrittlement, stress relief cracking, grain boundary corrosion and some kinds of intergranular stress corrosion cracking.[31,32] Basically, the segregation is closely related to the process of diffusion, in particular, the thermal diffusion segregation is most concerned due to the enhanced diffusion at high temperatures. In our knowledge, the atomic diffusion can be greatly enhanced and the thermodynamic barrier for phase transformations can be decreased when the electric current is applied to the metallurgy processes. Experiment has demonstrated a significant effect of electropulsing on the microstructural evolution in metals, e.g. segregation of lead inclusions in Cu-Zn alloy.[12] Here, the Fe-Cu model alloy with extremely low mutual solubility of copper in iron and cementite makes this an attractive system for investigating the basic segregation behavior under pulsed electric current.[33]

According to thermodynamics, the segregation increases with a decrease in free energy and with an increase in bulk concentration. In the solidification, a large bulk concentration of copper should have increased the segregation in the untreated alloy (e.g. thermal diffusion segregation). In the experiment, majority of the copper precipitates were dispersed in the matrix (**Figure 8**a and 8b). It suggested that energy barrier for segregation at grain boundaries should be much higher in this case, thereby retarded the segregation. Experimental results have confirmed that the copper precipitates prefer to segregate at dislocations and stacking faults within the matrix in the thermal diffusion segregation. The grain-boundary segregation of copper precipitates in pulsed alloy indicated that energy barrier for segregation at grain boundaries must be decreased (**Figure 8**c and 8d). When an electric current passes through a conductor, their system free energy encloses an additional term G_e in comparison with the system without electric current. Based on the Equation (4), $\Delta G_e < 0$ due to $\sigma_{Fe} < \sigma_{Cu}$, meaning that the free energy of segregation should be much negative due to an additional term. The segregation toward grain boundary in pulsed alloy increases with decreasing free energy and with increasing bulk concentration. In a word, the segregation is closely related to the process of diffusion, e.g., the segregation is exacerbated by the enhanced diffusion in the pulsed alloy.[33]

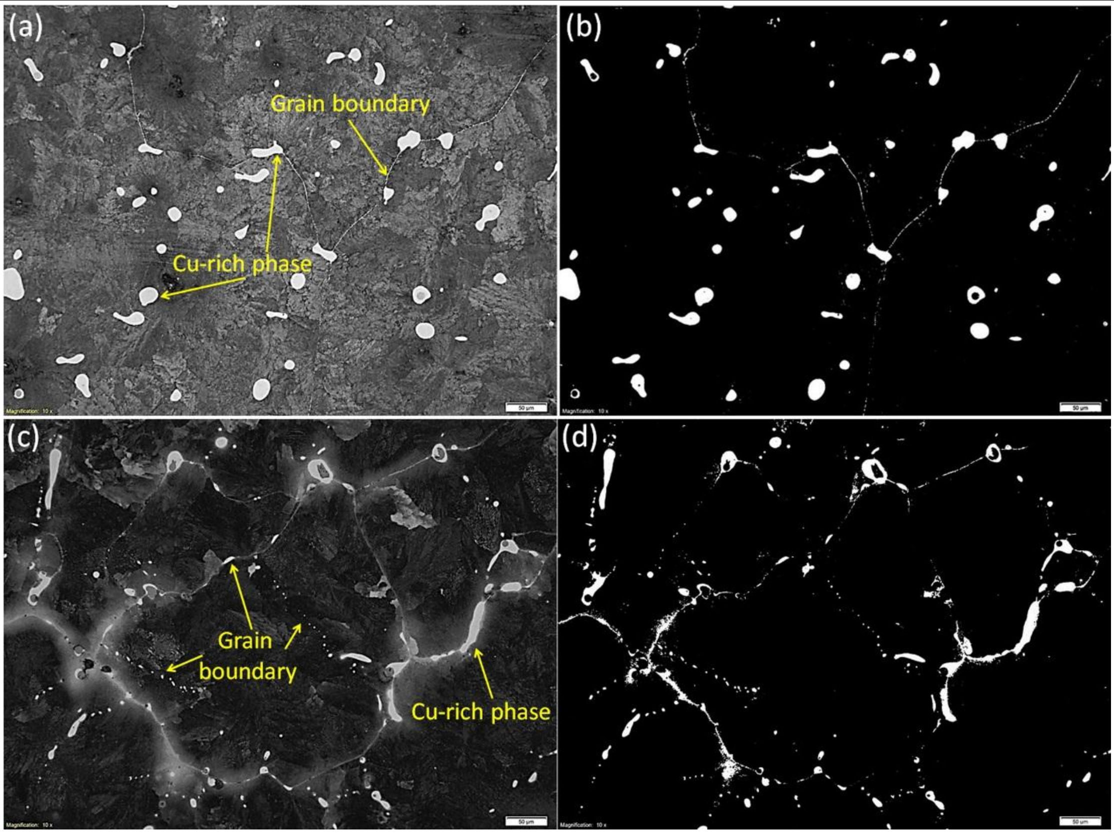

Figure 8. The distribution of copper precipitates before and after the pulsed electric current. (a, b) for the untreated alloy, and (c, d) for the treated alloy. Reproduced with permission.[33] Copyright 2015, Taylor & Francis Group.

3.4 Phase refinement under pulsed electric current

The mechanical properties of metallic materials are closely related to their microstructure, that is, the fine the microstructure, the strong the materials. By controlling the phase size to achieve modification on the performance of structural materials is a crucial part. For solid materials, mechanical plastic deformation and heat treatment (e.g. recrystallization) are commonly used for the grain refinement.[34,35] Here, we provide a new method called electropulsing to control the particle size. In the introduction section, we have introduced the possibility of refinement from the thermodynamic point of view. In this section, we use some experimental evidence to support the plausibility of thermodynamic predictions.

Figure 9. presents the refinement by passing electric current to a cold drawn pearlitic

steel rod at ambient temperature.[36,37] The strained cementite plates (Figure 9a) break up into nanoscale particles (Figure 9b) without change the integrity of materials. The microstructure is extremely fine and has not been reported obtainable using other conventional refinement methods.

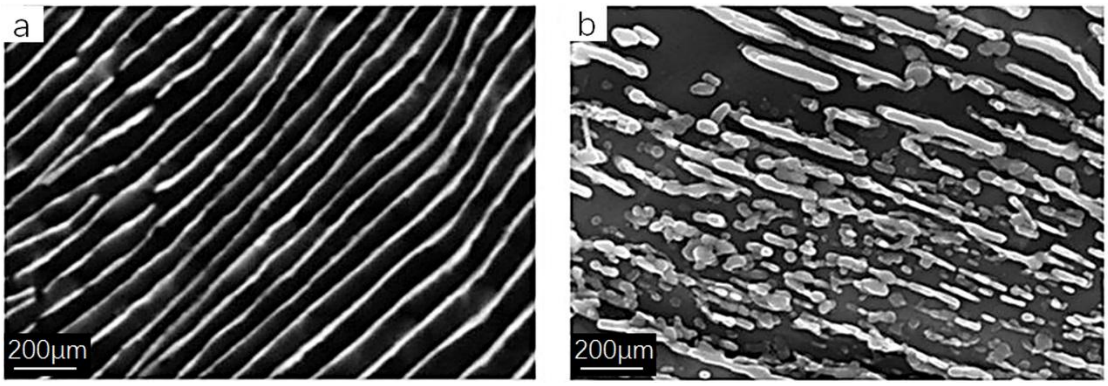

Figure 9. Scanning electron microscope images show the electric-current-driven Fe_3C stretched thin plates to break up into fine grains. (a) before electric current (b) after electric current. Reproduced with permission.[36] Copyright 2017, Springer Nature, Macmillan Publishers Limited.

It is also known that electric current helps to generate refined microstructure during liquid-liquid, liquid-solid and solid-solid phase transformation.[38,39] Electric current free energy plays an important role in those phenomena.

4. Urgent problems to be solved

Although some interesting and important experimental results have been found, the understanding on the mechanism for particle reconfiguration under external field is still not clarified yet. It is viable to explain the possibilities and trends for the above experimental phenomena based on thermodynamics, but there are bottlenecks in the understanding of particle reconfiguration from the kinetics. The rapid diffusion of atoms that occurs under an electric field can no longer be explained by the electromigration theory, since atom diffusion at electromigration is much less than this rapid diffusion. Combined with the current experimental results, the following points worth exploring:

(1) Such rapid diffusion usually occurs at a certain temperature, that is, the diffusion on external field is similar to that under the conventional heat treatment if a certain critical temperature can not reach. However, the diffusion of atoms in the electric field becomes very fast when the critical temperature reaches. This is why researchers are tangled in the dominant proliferation between Joule heating and electrical effects. From the electroplasticity of superconductors, the athermal effect (e.g. electrical effect) dominates the microstructure modification rather than the thermal effect.[40] Obviously, the electrical effect must exist, but when it plays a decisive role, it needs a certain temperature and other conditions.

(2) The solution to the thermodynamic equation is quite complex, especially in multiphase alloys, because its analytical boundary conditions and its approximation should not be the same as that in the single-phase systems.[10,15,41] At present, the solution to the thermodynamic equation is relatively simple because we consider all the events in the same conditions, which can not explain the phenomenon well.

(3) For the particle reconfiguration under external field, there is a serious shortage of research data. The data for particle manipulation in different kinds of environments are still very deficient. Based on the accumulated data, a reasonable solution to the thermodynamic equation is helpful to understand the particle reconfiguration mechanism under external field.

5. Prospect

Particle reconfiguration for the improvement of mechanical properties of metallic materials is of great significance. However, only the interaction mechanism for particles under external field is clarified, the accurate control of particle configuration in the complex environment can be realized. All the phenomena, including inclusion separation, element segregation, precipitate dissolution, and grain refinement, require more accurate data to discuss the theory and mechanism. The accumulation of research data on particle reconfiguration under external field is still on the way.

The work was financially supported by National Natural Science Foundation of China (51601011), Fundamental Research Funds for the Central Universities, Recruitment Program of Global Experts, and EPSRC (EP/J011460/2).

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Figure and Table Captions:

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